ANL-7820

EMICAL ENGINEERING DIVISION

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IANNUAL REPORT

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ARGONNE NATIONAL LABORATORY, ARGONNE, ILLINOIS

Printed in the United States of America
Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22151
Price: Printed Copy $3.00; Microfiche $0.95

ANL-7820
Metals, Ceramics,
and Materials

ARGONNE NATIONAL LABORATORY
9700 South Cass Avenue
Argonne, Illinois 60439

CHEMICAL ENGINEERING DIVISION

REACTOR SAFETY AND PHYSICAL PROPERTY STUDIES SEMIANNUAL REPORT

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July 1971

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ABSTRACT

A report of the work on Reactor Safety and Physical Properties Studies performed in the Chemical Engineering Division at Argonne National Laboratory is given for the period January through June 1971. Calorimetric studies include measurement of the enthalpy of Na_3UO_4 from 600°K up to about 1200°K and of $(U_{0.8}Pu_{0.2})O_{1.97}$ from 2348 to 3041°K. Partial infrared spectra for uranium oxide species in the region 1000-600 cm^{-1} have been obtained by matrix isolation spectroscopy. Work is in progress on the determination of the speed of sound in sodium from 1270 to 1800°K. Calculation of fission-product and fuel vapor pressures at high temperatures (~6000°K) based on extrapolation of available thermodynamic data is nearing completion. The electron-beam furnace to be used in determining the thermal diffusivity of molten UO_2 is being tested after completing required modifications of the furnace. Studies of the distribution of Zr, Ce, La, Y, Mo, and Ru between molten UO_2 and iron have been completed.

SUMMARY

Resistance-Heated Drop Calorimetric System

The compound Na_3UO_4 is important in the event of LMFBR fuel-pin failure, where sodium coolant would come in contact with the oxide fuel; high-temperature enthalpy increments have been obtained for a preliminary sample of this material, and further measurements are under way on a final sample.

Induction-Heated Drop Calorimetric System

Measurements have been made of the enthalpy of $(U_{0.8}Pu_{0.2})O_{1.97}$ in the temperature range from 2348 to 3041°K. Analyses of samples and data are still being performed. Preparations are being made for measurements of the enthalpy of liquid mixed-oxide materials.

Matrix-Isolation Spectroscopy

Accurate values of the thermodynamic properties of uranium oxide vapor species, which are needed for reactor-safety vapor-pressure calculations, can be obtained from spectroscopic parameters of these molecules. An experimental apparatus for trapping these species by the matrix-isolation technique has been built and successfully operated. Partial infrared spectra covering the region 1000-600 cm^{-1} have been obtained. Work is proceeding on the analysis of these spectra so that frequency assignments of the observed bands can be made.

Speed of Sound in Molten Reactor Materials

Work is currently in progress to extend measurements of the speed of sound in sodium from the previously reported high temperature of 1001°C up to 1500°C.

Theoretical Extrapolation of Measured Physical Property Data to Higher Temperatures

Calculations of fission-product and fuel vapor pressures have been modified to account for solubility of condensed fission-product oxides in the fuel lattice. The results obtained do not differ significantly from those previously reported, assuming mutual insolubility of the fission-product condensed phases at the relatively low temperatures (2500°K) for which the calculations have been carried out. Work on tabulation of thermodynamic data for fission-product species to 6000°K is near completion. At the higher temperatures, at which oxide partial pressures are more important, the results can be expected to reflect the changes in the calculational algorithm to a much larger degree.

Thermal Diffusivity of Reactor Materials

Values of the thermal diffusivity of UO_2 in the liquid state are of importance to the reactor safety program. The thermal conductivities of liquid UO_2 and of solid UO_2 at temperatures between 2000°C and the melting temperature of UO_2 are also required to help determine temperature distribution in the fuel. Thermal diffusivity will be determined from the phase change in a thermal wave passing through a sample; this wave is produced by heating the sample with a sinusoidally modulated electron beam. The thermal conductivity is calculated using the thermal diffusivity, heat capacity, and density. Modification of a twin-beam electron furnace for use in this work has been completed.

Reactor Materials--Fuel Phase Studies at High Temperature

Knowledge of the distribution of fission products to the phases which are produced in the event of a meltdown of the core of an LMFBR is required in order to predict the fission-product heat distribution after an accident. The information will be used to assure that means are available for cooling the products of the meltdown.

Studies of the distribution of inactive fission products between molten iron and molten UO_2 have been completed for some of the more important heat-producing elements. At a temperature between 2850 and 3000°C in an arc-melting furnace, Zr, Ce, La, Y, and Pr are distributed to molten UO_2. Mo and Ru are distributed to the molten-iron phase.

REACTOR SAFETY AND PHYSICAL PROPERTY STUDIES

The primary objective of these studies is to provide physical property data for use in evaluating the safety of various fast-breeder reactor materials. The property data obtained experimentally at temperatures above normal operating conditions will also be extrapolated to the much higher temperatures involved in accident situations. In addition, reactor material-fuel phase studies at high temperatures are under way to provide chemical information needed on fission-product distribution between molten fuel and other reactor materials for use in post-accident heat-removal calculations.

I. ENTHALPIES AND HEAT CAPACITIES BY DROP CALORIMETRY
(M. G. Chasanov)

Three complementary calorimetric systems for the measurement of high-temperature enthalpy increments are available for our use. Depending on the material to be studied and temperature range to be covered, one may choose (1) the resistance-heated drop calorimetric system (500-1600°K), (2) the electron-beam-heated drop calorimetric system (1300-2500°K), or (3) the induction-heated drop calorimetric system (2500-3600°K). During this period, systems (1) and (3) have been used for making measurements.

A. Resistance-Heated Drop Calorimetric System (D. R. Fredrickson)

The program for this system is oriented primarily toward obtaining physical property data for materials of interest in reactor technology, with a secondary interest in materials useful in high-temperature technology.

1. Na_3UO_4

The compound Na_3UO_4 has been shown to form in the event of a LMFBR fuel-pin cladding failure when the sodium coolant comes in contact with uranium dioxide fuel. Thus, this material currently is of considerable interest in fast-reactor technology. High-temperature enthalpy data have been obtained by us on a compound with the nominal composition Na_3UO_4 over the temperature range from 600 to 1200°K. This data is needed for computation of thermodynamic functions for the material in order to evaluate its possible reactions and range of stability.

A preliminary sample (FS-32) of Na_3UO_4 was used for these measurements. Based on sodium and uranium analysis, this sample contains 90.3 mol % Na_3UO_4 and 9.7 mol % UO_2. The experimental data are shown plotted in Fig. 1. This is a plot of total heat measured (in calories) vs. the temperature (in degrees Kelvin) for a series of eighteen drops. The measured heat can be assigned as follows: 55% due to the sample, 42% due to the tantalum capsule, and 3% due to the UO_2 impurity. Figure 1 is intended primarily to show the precision of the results. As can be seen, the calorimetry is adequate, but the exact characterization of the

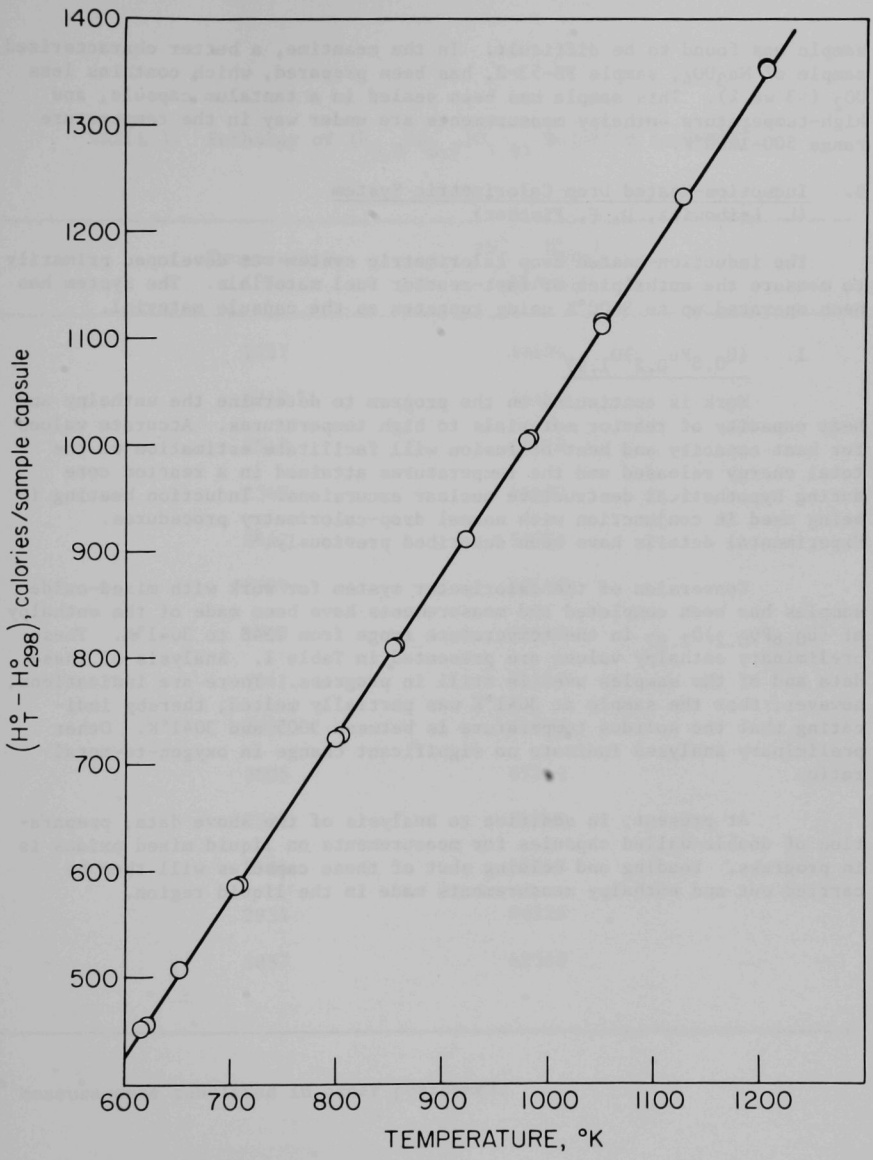

Fig. 1. Measured Enthalpy for Na-U-O Sample (Sample FS-32) Plus Capsule as a Function of Temperature

sample was found to be difficult. In the meantime, a better characterized sample of Na_3UO_4, sample FS-53-2, has been prepared, which contains less UO_2 ($\sim$3 wt %). This sample has been sealed in a tantalum capsule, and high-temperature enthalpy measurements are under way in the temperature range 500-1200°K.

B. Induction-Heated Drop Calorimetric System
(L. Leibowitz, D. F. Fischer)

The induction-heated drop calorimetric system was developed primarily to measure the enthalpies of fast-reactor fuel materials. The system has been operated up to 3600°K using tungsten as the capsule material.

1. $(U_{0.8}Pu_{0.2})O_{1.97}$

Work is continuing on the program to determine the enthalpy and heat capacity of reactor materials to high temperatures. Accurate values for heat capacity and heat of fusion will facilitate estimation of the total energy released and the temperatures attained in a reactor core during hypothetical destructive nuclear excursions. Induction heating is being used in conjunction with normal drop-calorimetry procedures. Experimental details have been described previously.[1]

Conversion of the calorimeter system for work with mixed-oxide samples has been completed and measurements have been made of the enthalpy of $(U_{0.8}Pu_{0.2})O_{1.97}$ in the temperature range from 2348 to 3041°K. These preliminary enthalpy values are presented in Table 1. Analysis of these data and of the samples used is still in progress. There are indications, however, that the sample at 3041°K was partially melted, thereby indicating that the solidus temperature is between 3005 and 3041°K. Other preliminary analyses indicate no significant change in oxygen-to-metal ratio.

At present, in addition to analysis of the above data, preparation of double-walled capsules for measurements on liquid mixed oxides is in progress. Loading and welding shut of these capsules will then be carried out and enthalpy measurements made in the liquid region.

TABLE 1. Enthalpy of $(U_{0.8}Pu_{0.2})O_{1.97}$ Relative to 298°K[a]

Temperature (°K)	$(H_T^\circ - H_{298}^\circ)$ (cal/mol)
2427	46694
2718	56450
2518	49528
2348	44497
2642	53564
2799	59112
2969	65548
3041	71160
2892	61983
3005	67463
2572	51049
2934	64149
2934	64125
2887	62368

[a] Measurements tabulated in order performed.

II. MATRIX ISOLATION SPECTROSCOPY
(M. G. Chasanov)

The thermodynamic properties of the vapor species of fuel oxides are important in determining equation-of-state relationships used in projecting the results of certain fast-breeder-reactor design-basis accidents. Application of statistical mechanical techniques to spectroscopic data for these species presents a useful way of determining their thermodynamic properties at temperatures beyond the range of direct experimental measurements. Consisting of trapping molecules in frozen inert-gas lattices, the matrix isolation method allows low-temperature pseudo-gas phase studies to be carried out on molecules that would normally be present in high gas-phase concentrations only at very high temperatures. Because the population of excited rotational quantum levels is eliminated under these low-temperature conditions, interpretation of the simplified infrared and visible spectra is greatly facilitated.

A. UO_2 (S. D. Gabelnick, G. T. Reedy)

Estimates of the relative magnitudes of fission-product and fuel vapor pressures in closed systems indicate that, for initial concentrations of fission products approximating those found in fuel pins at 3-10% burnup, the vapor pressure of fuel species begins to dominate the total pressure above 5000°K. This qualitative conclusion points to the necessity of measuring or calculating the thermodynamic properties of fuel species to high accuracy when assessing the role of vapor pressure in design-basis accidents involving fuel temperatures above 4000-5000°K. In addition to extrapolating thermodynamic data of the condensed fuel, one must know the free energies of formation of the species in equilibrium with the condensed fuel in order to determine the overall pressure. Tabulations of free-energy functions of uranium oxide species, such as those appearing in Schick's compendium,[2] have, in general, been based on spectroscopic quantities estimated by De Maria *et al.*[3] The objective of this program is to improve the values of the thermodynamic functions of fuel vapor species by direct measurement of the spectroscopic properties from which the thermodynamic properties can be calculated using statistical mechanics.

A portion of the infrared spectrum of oxides of uranium trapped in an argon matrix is shown in Fig. 2. Nearly stoichiometric $UO_{2\pm x}$ was heated in a tungsten Knudsen cell to ∿1900°C and allowed to condense with a stream of argon on a KBr window at ∿16°K. The spectrum was obtained using an infrared Fourier transform spectrometer system after deposition for 2 hr. Examination of the spectrum shows strong absorptions at 746, 776, and 853 cm^{-1} and weaker absorptions at 801, 818, and 871 cm^{-1}. Current work includes the assignment of these absorptions to the appropriate uranium oxide vapor species. This can be accomplished by variation of the relative concentrations of the different species trapped in the matrix by changing the O/M ratio of the condensed material in the Knudsen cell and following the resultant changes in peak

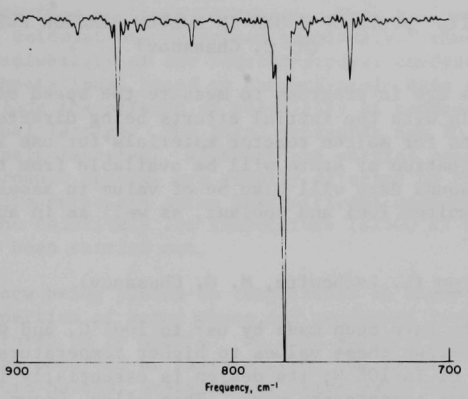

Fig. 2. Portion of Infrared Spectrum of Uranium Oxide Species Trapped in an Argon Matrix

intensities. Assignment can be further aided by normal-coordinate analysis of the above observed frequencies and the frequencies of species containing ^{18}O when the frequencies are measured to high accuracy. Present instrumental capabilities allow measurement of these frequencies to better than ± 0.1 cm^{-1}, which should be adequate for such analyses and, in addition, for the determination of such important parameters as the bond angle of $UO_2(g)$ by the isotope-shift technique.[4]

III. SPEED OF SOUND IN MOLTEN REACTOR FUELS
(M. G. Chasanov)

Experiments are in progress to measure the speed of sound in molten reactor materials with the initial efforts being directed to sodium. Compressibility data for molten reactor materials for use in conjunction with the Gruneisen equation of state will be available from these measurements. These speed-of-sound data will also be of value in assessing shock propagation through molten fuel and coolant, as well as in analyzing two-phase flow of coolant.

A. Molten Sodium (L. Leibowitz, M. G. Chasanov)

Measurements have been made by us[5] to 1001°C, and work is currently in progress to extend these values to higher temperatures. A new cell has been fabricated of Ta-10% W; its design is essentially identical to that used for the lower temperature work. This alloy, known as 60 Metal, has the strength and sodium-resistant properties needed in these experiments. The completed cell has been tested by measuring the speed of sound in mercury and in ethyl alcohol; the measured values agree to within about 0.1% of literature data. The Varian furnace, which will be used in this work, has been tested extensively and is operating satisfactorily. The cell will shortly be loaded with sodium and measurements performed to about 1500°C.

IV. THEORETICAL EXTRAPOLATION OF MEASURED PHYSICAL PROPERTY DATA TO HIGHER TEMPERATURES
(M. G. Chasanov)

The high-temperature experimental studies reported in the preceding sections can provide physical property data for fuel materials, cladding, coolant, and structural materials at temperatures that are extremely high from our experimental point of view ($\sim$3300°K) but are still markedly less than temperatures of interest in reactor safety analyses. To satisfactorily correlate and extrapolate experimental data for utilization at these significantly higher temperatures ($\sim$6000°K), recourse to existing theory, such as corresponding-states, scaled-particle, and significant-structures, is necessary. Our program involves calculational studies, based on theory, to assess consistency and validity of such extrapolation and correlation of available data.

A. Fuel and Fission-Product Vapor Pressures
(S. D. Gabelnick, M. G. Chasanov)

Data on the vapor pressure over fuel/fission-product systems at high temperatures are needed for input to the disassembly codes describing core movement and fuel-failure propagation in certain types of fast-breeder-reactor design-basis accidents. Because actual pressure measurements are generally unavailable for these systems at high temperatures (3000-6000°K), an effort has been made to calculate such pressures based on the available thermodynamic properties of these materials. Work has continued on the refinement of the calculations described previously;[6] those calculations assumed mutual insolubility of the fission-product condensed phases. Results of the computations, based on thermodynamic data of Bedford and Jackson,[7] now include the effects of solubility of condensed oxide fission products in the fuel lattice. The total pressures are 30 to 50% lower than those previously reported. Although the partial vapor pressures of certain fission-product species are greatly affected by the program modifications, these pressures are often insignificant compared with the major contributors at the relatively low temperature ($\lesssim$2500°K) for which the calculations have been carried out.

Emphasis is now being placed on compilation of the self-consistent thermodynamic properties of vapor phase and condensed fission-product oxides at temperatures up to 6000°K. No such satisfactory compendium of thermodynamic data, observed or extrapolated, now exists. Above 4000°K, at which temperature partial pressures of oxide vapor species begin to predominate, the calculated pressures should become more sensitive to parameters such as initial fuel composition and deviation from unit activities of condensed oxides dissolved in the fuel lattice. Satisfactory results from this type of calculation should be available for the range 3000-6000°K upon conclusion of the tabulation of the appropriate thermodynamic data.

V. THERMAL DIFFUSIVITY OF REACTOR MATERIALS
(M. G. Chasanov)

Values of the thermal diffusivity of reactor materials in the liquid state are needed for the reactor safety program to evaluate means of safely cooling the core of an LMFBR in the event of a meltdown. The thermal diffusivity can be used in conjunction with heat-capacity and density data to provide thermal conductivity data for oxide fuel, which is needed to help in determining temperature distribution in fuel, cooling rates, and heat dissipation and in establishing suitable containment measures for molten fuel in a reactor meltdown accident.

A. Liquid UO_2 (J. Fischer, L. Leibowitz, J. Haley)

Initially, a reliable experimental technique must be developed for measurement of thermal diffusivity of liquid materials at high temperatures. This technique will first be applied to UO_2 and mixed-oxide fuels. The UO_2 measurements will extend from about 1200°C, where there is agreement among various investigators, through the upper limit of previous

measurements (2100°C) to temperatures above the melting point of UO_2, with special attention to temperature dependence at the higher temperature regions. The program will then be expanded to include other reactor materials, *e.g.*, other fuels and cladding.

In the experimental method, which has been described previously,[8] thermal diffusivity is determined from the phase change in a thermal wave passed through a sample. This wave is produced by heating the sample with a sinusoidally modulated electron beam. A twin-electron-beam furnace, formerly used as a tensile-testing apparatus and modified for the thermal-diffusivity measurements, will be employed. Modification involved repositioning the electron-beam guns in the vertical position instead of the horizontal position used for the tensile tests, placing four additional viewing ports on the furnace chamber, installation of rotatable sample positioners in the chamber, and installation of radiation shields and supports inside the furnace chamber. The electron guns were replaced since they were of a very old design and not satisfactory for this problem. Various electronic modifications have been made on the system, and a phase-shift measurement apparatus developed and tested. Tungsten sample containers have been fabricated and will soon be tested for use with molten UO_2, and a preliminary evaluation of the effect of beam-scanning frequencies on the tungsten surface has been completed. Testing of the system is still in progress.

VI. REACTOR MATERIALS -- FUEL PHASE STUDIES AT HIGH TEMPERATURES (M. G. Chasanov)

Knowledge of the distribution of fission products to the phases produced in the event of an LMFBR core meltdown is required to predict the fission-product heat distribution after an incident and be able to assure that means are available for cooling the products of the meltdown. In the type of hypothetical incident being considered, simultaneous failure of multiple safeguards and loss of effective cooling could lead to melting of the oxide fuel, the stainless steel cladding, the core-support steel, and the concrete container. Consideration must also be given to the possible dissolution of concrete in the molten UO_2-PuO_2 layer, thereby forming a liquid less dense than molten steel. To properly evaluate the capabilities of heat removal after a meltdown incident, it is necessary to know the physical distribution of the phases; also required is the quantitative distribution of fission-product heat among the phases produced. This program seeks to provide this information.

A. Distribution of Fission Products among Molten Fuel and Reactor Material Phases (J. Fischer, J. Schilb)

The objective of these studies is to investigate the phase interactions that result when molten fuel materials are in contact with cladding, structural materials, coolant, and containment materials. The determination of the nature of immiscible phases and the distribution of important decay-heat-generating fission products between the metallic and oxide phases possible in a meltdown situation will be included in these

studies. A study of the composition of the liquidus and the melting points of equilibrium phases for important components of cladding, structural material, and containment materials with oxide fuel will be determined in those cases where insufficient information is currently available.

Studies of the distribution of inactive fission products between molten iron (representing fuel cladding) and molten UO_2 (representing reactor fuel) have been completed using an arc-melting furnace. The experimental method has been previously described.[9] The distribution of some of the more important decay-heat-generating fission products has been studied. These fission products are Zr, Ce, La, Y, Pr, Mo, Ru, and Nb. The analytical results, which include colorimetric, mass spectrometric, X-ray fluorescence, and microprobe assays, have now been obtained for all of the elements studied, with the exception of Nb. Additional analytical information will be obtained for Nb. An ANL topical report is being prepared which will contain all of the information in detail. The experimental data to date are summarized in Table 2.

In order to establish that sufficient time was allowed for reaction and distribution of the fission products between the phases, equilibrium was approached from more than one direction for several of the fission products--Zr, Mo, and Ru. Because the oxides of Ce, La, Y, and Pr have marked thermodynamic stability at the experimental conditions, and their behavior should be similar to zirconium, the reactions for these elements were carried out with the fission products as oxides in the initial state. Because there is variable loss of UO_2 as vapor and a small loss of iron due to volatility under the experimental conditions, no attempt was made to make material balances for the phases or the fission products. The extent of fission-product distribution is evident in the concentrations of the components in the oxide or metal phases after reaction. There is some inclusion of each phase in the other after the phases have been cooled. Microprobe analysis of cross sections of the phases for the fission-product elements, iron, and uranium has clearly indicated that, when the concentrations of F.P. in a phase are very low, this is due to particulate inclusion and not representative of the true distribution in the bulk phase.

It is evident from the results in Table 2 that Zr, Ce, La, Y, and Pr are distributed to the molten UO_2 and that Mo and Ru are distributed to the molten iron phase. These results, for the most part, support the conclusions drawn from available thermodynamic data. To date, inconclusive results have been obtained for Nb because of incomplete analytical data.

Design and testing of an isothermal apparatus, which will be used to determine the distribution of fission products between a molten UO_2-concrete mixture have been completed. The most important aspect of this part of the work was to establish that a zirconium silicate crucible is a suitable container. Various crucible materials were extensively tested before zirconium silicate was chosen. Work on determining the solubility of UO_2 in a simulated, molten, concrete mixture is now in progress, and it

TABLE 2. Distribution of Inactive Fission Products (F.P.) Between Molten Iron and UO_2 (Temp. 2850-3000°C[a])

Fission Product, Initial Form	Original Phase[b]		Final Phase[c] F.P. Element (wt %)	
	Phase	F.P. Element (wt %)	UO_2	Fe
Zr	Fe	0.90		<0.03[d]
ZrO_2	UO_2[e]	1.00		<0.04[d]
CeO_2	UO_2	0.82	0.68[f]	0.02[d]
La_2O_5	UO_2	0.26	0.35[g]	<0.3 ppm[g]
Y_2O_3	UO_2	0.79	1.5[h]	0.04[d]
PrO_2	UO_2	0.82	1.4[h]	0.02[d]
Mo	Fe	1.10	(0.12)[i]	1.20
MoO_3	UO_2	4.50	0.12 (0.17)[i]	1.06
Ru	Fe	1.02	(0.0)[i]	1.14
RuO_2	UO_2	0.76	<0.1	0.61

[a] Between the melting point of UO_2 and the boiling point of Fe.

[b] Elemental concentration of F.P. in oxide phase was calculated for mixture. Concentration of F.P. in metal was determined by colorimetric assay of Fe-F.P. alloy before equilibration.

[c] After equilibration for 6 to 12 min at 2850-3000°C.

[d] Low concentration of F.P. in the metal phase (by colorimetric assay) due to inclusions of small particles of UO_2 in the solidified Fe phase, which was verified by microprobe X-ray analysis. Less than values (<) are the lower limits of detection for the assay.

[e] All starting oxide phases were mixtures of the F.P. oxides and UO_2.

[f] Colorimetric assay after removal magnetically of the bulk of the included iron from the oxide phase after it was ground up.

[g] Spark source mass spectrometric analysis.

[h] Higher concentration of F.P. in the UO_2 phase after equilibration is due to loss of UO_2 by volatilization and concentration of the F.P. Amount of UO_2 vaporized varied for each experiment.

[i] Estimated from microprobe analysis, which showed that the Mo and Ru values were concentrated at points of Fe inclusions in the UO_2 phase.

appears that the solubility of UO_2 is between 7 and 10 wt % in a concrete mixture typical of that used in reactor structures.

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B